PB88-170576

Calcium Hydroxide and Calcium Carbonate Particle Size Effects on Reactivity with Sulfur Dioxide

Acurex Corp., Research Triangle Park, NC

Prepared for

Environmental Protection Agency
Research Triangle Park, NC

1987

U.S. Department of Commerce
National Technical Information Service
NTIS

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completir

1. REPORT NO.	2.	3. PB88-170576
EPA/600/J-87/210		

4. TITLE AND SUBTITLE
CALCIUM HYDROXIDE AND CALCIUM CARBONATE PARTICLE SIZE EFFECTS ON REACTIVITY WITH SULFUR DIOXIDE (Journal Version)

5. REPORT DATE
1987

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
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8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Acurex Corporation
P.O. Box 13109
Research Triangle Park, North Carolina 27709

10. PROGRAM ELEMENT NO.

11. CONTRACT/GRANT NO.
68-02-3988

12. SPONSORING AGENCY NAME AND ADDRESS
EPA, Office of Research and Development
Air and Energy Engineering Research Laboratory
Research Triangle Park, NC 27711

13. TYPE OF REPORT AND PERIOD COVERED
Journal article; 10/85-9/86

14. SPONSORING AGENCY CODE
EPA/600/13

15. SUPPLEMENTARY NOTES AEERL project officer is Brian K. Gullett, Mail Drop 4, 919/541-1534. Ref: Reactivity of Solids, 3:337-350, September 1987

16. ABSTRACT

The paper reports results of measurements of the effect of in situ calcium-based sorbent particle size upon reactivity with 3000 ppm SO2 in an 1100 C drop-tube furnace, using on-line collection of the reacted sorbent with a particle cascade impactor. Significant agglomeration occurred for Ca(OH)2, while little was apparent for CaCO3. Reactivity of all sorbent particles increases as the measured particle diameter decreases, such that conversion is inversely related to diameter to the 0.22 to 0.32 power. For these sorbents, Ca(OH)2 particles had greater reactivity than the similarly sized CaCO3 sorbent.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Sulfur Dioxide	Stationary Sources	07B
Reactivity		07D
Particle Size		14G
Sorbents		11G
Calcium Hydroxides		
Calcium Carbonates		

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL
INFORMATION SERVICE
SPRINGFIELD, VA. 22161

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
Release to Public	Unclassified	17
	20. SECURITY CLASS (This page) Unclassified	22. PRICE A03 12.95/A01 6.95

EPA Form 2220-1 (9-73)

EPA/600/J-87/210
JOURNAL ARTICLE

Reactivity of Solids, 3 (1987) 337–350
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

CALCIUM HYDROXIDE AND CALCIUM CARBONATE PARTICLE SIZE EFFECTS ON REACTIVITY WITH SULFUR DIOXIDE *

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(Received December 6th, 1986; accepted February 2nd, 1987)

ABSTRACT

The effect of in situ calcium hydroxide [$Ca(OH_2)$] and calcium carbonate ($CaCO_3$) sorbent particle size upon reactivity with 3000 ppm sulfur dioxide (SO_2) in an 1100 °C drop-tube furnace was measured by on-line collection of the reacted sorbent with a particle cascade impactor. Significant agglomeration occurred for the hydroxides, while little was apparent for the carbonate. Reactivity of all sorbent particles increases as the measured particle diameter decreases such that conversion is inversely related to diameter to the 0.22–0.32 power. For these sorbents, $Ca(OH)_2$ particles had greater reactivity than the similarly sized $CaCO_3$ sorbent.

INTRODUCTION

The cost-effective use of calcium-based sorbents for removal of sulfur dioxide (SO_2) gas by dry injection into utility coal boilers is dependent upon minimizing sorbent costs by improving reactivity. Current results [1] indicate that under optimal conditions calcium hydroxide sorbents [$Ca(OH)_2$] achieve greater removal of SO_2 than calcium carbonate ($CaCO_3$) sorbents. The $Ca(OH)_2$ sorbents are derived from hydrating calcium oxide (CaO), the calcination product of $CaCO_3$. This increased reactivity is thought to be mainly due to the smaller particle size resulting from the hydration process. The smaller particle sizes are likely to be more reactive because they have greater surface area per volume and the resistance to diffusion of SO_2

through the pore structure is minimized. Crushing/grinding of $CaCO_3$ particles to a comparable size is commercially impractical [2], and so most attention has been focused on the use of hydrated sorbents.

Studies of particle size effects have traditionally used methods of gravity sedimentation and coulter counter analyses with dispersants to determine a sorbent's size distribution. These pre-reactor size distributions have then been qualitatively related to a sorbent's comparative reactivity [3]. This approach is inadequate in that hydrates have an extreme propensity to stick together, forming agglomerates. These agglomerates may react quite differently from their individual particles, and so attempts to relate a dispersed-sorbent size distribution to whole-sample reactivity are insufficient.

Another approach toward determining particle size effects upon reactivity has been to size sorbents to narrow diameter cuts prior to reactor injection and then determine the comparative reactivity of each diameter cut. This technique has been applied to carbonates with some success [4], but uncertainties between relating the cut material to the in situ size and difficulties of obtaining narrow size cuts remain. This method is especially difficult for calcium hydroxides because they readily reagglomerate after sizing, and their small mass median size makes narrow diameter cuts quite difficult to obtain.

Reactivity measurements have been made by Ishihara et al. [4] on limestones sized by screening prior to injection into an entrained flow tower reactor at 1100°C. Conversion to calcium sulfate ($CaSO_4$) was found to approximately double when the mean particle size decreased from about 50 to 4 μm. Two limestones tested produced different reactivities, but for both sorbents the conversions were inversely proportional to the 0.28 power of particle size. They hypothesized that the smaller particles were more reactive since their specific surface area was higher.

The question arises as to whether there is a limit to particle reactivity with decreasing size. Does the reaction rate approach an asymptote with smaller particles, a case which represents loss of diffusion control and transfer to chemical reaction control? Or does pore diffusion control continue to be important even as particle size decreases?

This present research continues measurement of particle size effects on reactivity but focuses mainly on $Ca(OH)_2$ performance, while a $CaCO_3$ sorbent is examined for comparative purposes. Techniques are used that measure the in situ, effective particle size of the sorbents, rather than relying on pre-reactor sorbent sizing. More specifically, three calcium hydroxides are tested: two that are produced commercially (one under atmospheric pressure and one under higher pressure) and a third produced in the laboratory with a water slaking technique. One $CaCO_3$ sorbent is tested. Measurements of particle size distribution are made by means of an inertial cascade impactor, and sorbent reactivity in a graphite element drop-tube furnace is determined by solids analysis with atomic adsorption and ion chromatography. Because the distinction of discrete particles from ag-

glomerates is vague, this paper will loosely define the agglomerate size as the in situ sorbent size and the constituent particle size as the solid dimensions that roughly include all porosity below 500–600 Å.

The objectives of this research were to determine the effect of agglomeration upon in situ particle size and to measure the particle size effect upon comparative reactivity of calcium-based sorbents, principally $Ca(OH)_2$. The former was accomplished by multiple determinations of particle size distribution with an inertial impactor measuring particle size both prior to and after furnace injection of the sorbent. The latter was met by analyzing the inertial impactor stages for Ca^{2+} and SO_4^{2-} after furnace injection.

The results of this study should provide experimental evidence of the influence of in situ sorbent particle size upon reactivity, and hence, may determine a practically achievable upper limit for SO_2 removal. The effect of particle agglomeration will be examined, and its impact upon sorbent reactivity determined. These results will have implications for sorbent choice, desired particle size for injection, understanding of physical mechanisms, and predictive models for the sorbent sulfation reaction.

EXPERIMENTAL

Sorbents

Four sorbents were used for this research: three $Ca(OH)_2$ sorbents and one $CaCO_3$ sorbent. Of the three calcium hydroxides, two were produced commercially by the Linwood Lime and Cement Co. These were produced by hydration under either atmospheric or higher pressures and are referenced as LAH and LPH, accordingly. The carbonate (FWL) was Fredonia White limestone, from Fredonia Quarries (KY, U.S.A.). The third hydroxide (FWAH) was prepared from an aerodynamically fractionated size cut of FWL with a narrow size distribution around a mass median diameter of 3.1 μm. This cut material was calcined in 3-g batches in a nitrogen-swept muffle furnace at 982°C for 90 min. The resulting CaO was then cooled under vacuum and transferred to a filter flask where it remained under positive nitrogen flow. A volume of 10 ml of 95°C water was added to the flask, and the slaked sorbent was dried overnight under flowing nitrogen. The molar ratio of water to CaO was 13:1, whereas for typical commercial hydration operations the ratio is around 2:1 [5]. The four sorbents were analyzed for their Brunauer-Emmet-Teller (BET) surface area by nitrogen sorption techniques. A rough estimate was made of the sorbents' median particle size by determining pore size at the end of the interparticle void region by mercury porosimetry. An upper limit for the corresponding particle size was calculated based upon hexagonal close packing of spherical particles with a

TABLE 1

Surface area and particle size of raw test sorbents

Sorbent	BET surface area (m^2/g)	Particle size (μm)
LAH	15.5	1.3
LPH	8.0	1.1
FWAH	45.6	1.2
FWL	1.8	5.2

particle to pore diameter ratio of 6.45 : 1.00. The results are shown in Table 1.

Impactors

Two inertial cascade impactors were used for sorbent particle sizing and reactivity measurements. Inertial impactors separate particle sizes by aerodynamic classification. The particle-laden gas stream passes through a series of stages with progressively smaller orifices that separate out particles with sufficient inertia to impact and remain on each stage. A Meteorological Research Incorporated (MRI) inertial cascade impactor was used for determination of the particle size distribution after ambient and high temperature sorbent environments. The median diameter (D_{50}) range of particles collected by the MRI stages ranged from 0.52 to 16.5 μm, while the first stage collected all particles with a D_{50} larger than 33.5 μm. A Gelman inertial impactor was used for sulfation analysis by particle size because the heated impactor could be operated without temperature-sensitive O-rings. The particle size for which sulfation data were obtained with the Gelman impactor ranges from 0.44 to 7.8 μm, with the pre-impactor stage collecting all particles with a D_{50} larger than 12.1 μm.

Reactor system

Sorbents were exposed to SO_2 in the high-temperature graphite element furnace reactor shown in Fig. 1, and described more fully elsewhere [6]. Essentially, the reactor serves as a drop-tube reactor into which the sorbent and SO_2 are fed, and then the sorbent is captured by an air-cooled quartz probe after leaving the reaction zone.

The feeder system (Fig. 2) is designed to uniformly feed sorbents by partially fluidizing the sorbent bed with a small fraction of the process gas. The particles are entrained in this carrier gas and swept into the fine-gauge tube from which they are injected axially into the reaction zone. A vibrator attached to the system agitates the sorbent reservoir, preventing channeling while maintaining sample homogeneity.

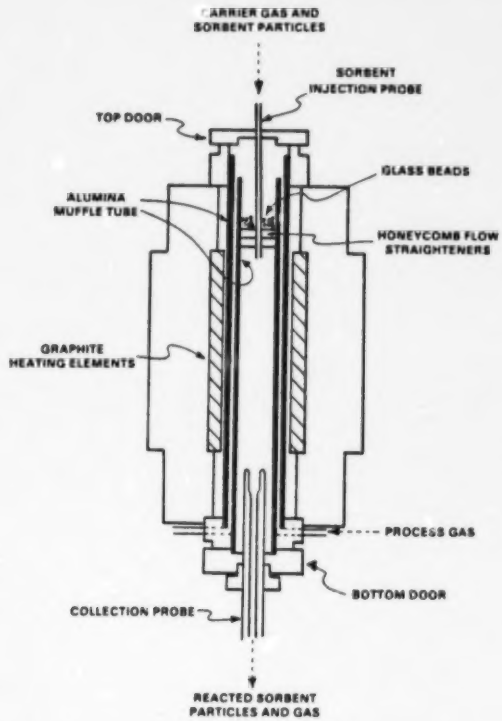

Fig. 1. Graphite element furnace reactor.

The quartz collection probe is air-cooled to provide a rapid quench of the sorbent reactions. The inertial impactors are connected below the collection probe to capture the injected sorbent.

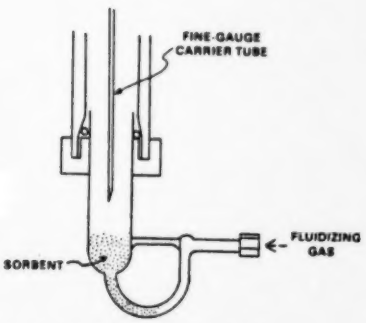

Fig. 2. Sorbent feeder system.

Procedure and analysis

For particle size distribution analysis, sorbent was fed through the reactor and into the collection probe at ambient temperatures, unless otherwise noted. From two sorbent tubes holding ca. 0.5 g each, nominally 250 mg of sorbent was fed in 30 min to constitute a single size distribution test. Four replicate determinations were averaged for the plotted values. The weight change of the impactor stages indicated the amount of sorbent collected for a particular size.

For sulfation analysis by size, sorbents were injected into the graphite element furnace reactor, passed through the collection probe, and captured on the impactor substrate trays. Four sorbent tubes were used to feed approximately 500 mg of sorbent in a total time of 60 min to constitute one sulfation analysis. At least eight of these analyses were averaged for the hydroxides and four for the carbonate to determine the single values of reactivity for each sorbent size. The reactor was maintained at a nominal temperature of 1100 °C with the process gas of 3000 ppm SO_2, 5% oxygen, and the balance nitrogen at a flow-rate of 9.2 l/min for a nominal residence time of 1.5 s. Sorbent was injected into the furnace at a rate of 5–10 mg/min, resulting in a steady-state molar ratio of sulfur to calcium within the reactor of around 9 : 1.

The impactor stages were analyzed for extent of reaction by determining the concentration of calcium ions, Ca^{2+}, and sulfate ions SO_4^{2-}. The Ca^{2+} concentration was determined by atomic absorption spectroscopy, and the SO_4^{2-} was analyzed by ion chromatography.

Precautions were taken to ensure that all of the measured conversion occurred as $CaSO_4$ formation in the hot zone of the reactor, and not due to reaction in the feeder assembly or on the impactor trays. Formation of SO_3^{2-} within a reactor has been noted previously [7]. This SO_3^{2-} can condense on cooled parts of the collection system, introducing sampling error. The impactor collection system was heated to 320 °C with mantles and heat tape to ensure that any SO_3^{2-} formed would not condense and impact upon the trays, altering the results toward higher conversions. Tests for SO_3^{2-} deposition on the impactor trays confirmed that the heated impactor eliminated all but scant traces of SO_3^{2-}.

In further tests, the SO_2–N_2–O_2 process gas flow was continued for 40 min more after the sorbent feeding was completed. The effect of more than doubling the mean solids residence time in the impactor was to increase conversion on each tray by about 5–20%. Thus, for regular testing durations, less than 2–8% conversion might be expected due to sorbent reaction on the impactor trays. Further, this reaction was not preferential to particle size, thereby preserving the high temperature sorbent size-conversion trends.

RESULTS

Sorbent size testing

Fig. 3 shows the sorbent size distribution of LAH determined by gravity sedimentation and by inertial impactor analysis. Dispersant techniques used with gravity sedimentation break up particle agglomerates, resulting in a much finer size distribution than that provided by the reactor feeder system. A scanning electron micrograph (Fig. 4) confirms that the larger particles appear to be formed by agglomerates of much smaller constituent particles.

To underscore this latter point, LAH was centrifugally size-classified in a Donaldson Acucut classifier. The size fraction less than 2.0 μm was collected and fed into the inertial impactor. The resulting size distribution (Fig. 5) is even larger than the original unclassified material, suggesting that the agglomerates are composed of particles that are smaller than 2.0 μm and that the smaller hydroxide particles are even more prone to agglomerate into larger particles.

The effect of high reactor temperatures upon the formation of hydroxide agglomerates was examined by comparing the sorbent size distributions resulting from feeding LAH through the heated graphite element furnace reactor (nominally 1100 °C) and the unheated reactor (25 °C). The two size distributions (Fig. 6) are indiscernible within the range of the experimental error. The high temperature does not seem to prevent the formation of hydroxide agglomerates or break them up.

The effect of SO_2 reaction upon sorbent size is also minimal. Fig. 7a shows the size distributions of LAH run through the reactor at a nominal temperature of 1100 °C, one set of data with SO_2, and one without. Within

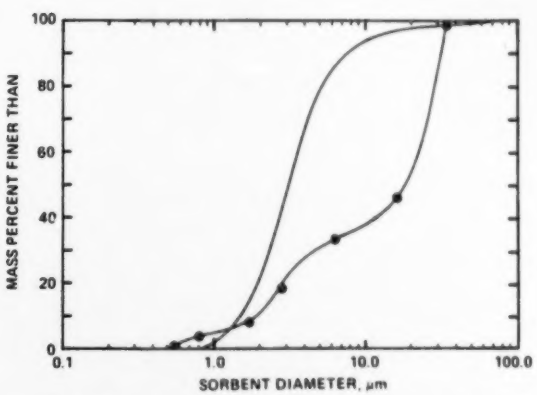

Fig. 3. Sorbent size distribution of LAH from gravity sedimentation (——) and the inertial impactor (●).

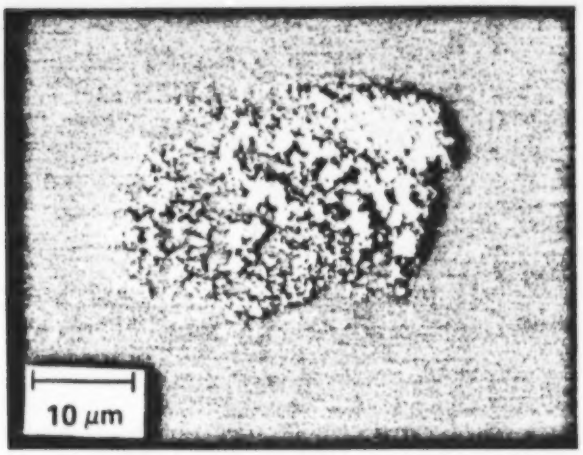

Fig. 4. Scanning electron micrograph of a hydrate agglomerate.

the precision of the impactor analysis, the formation of $CaSO_4$ product in the one set does not seem to alter the sorbent size. This effect is also consistent with another sorbent: LPH did not show any significant effect of SO_2 upon sorbent size distribution (Fig. 7b).

The differences between the size distributions of all four sorbents are shown in Fig. 8. Comparison of the calcium hydroxide sorbents to determine if hydration conditions affect sorbent size indicates little distinction between the commercial Linwood atmospheric and pressure hydroxides. The laboratory slaked hydroxide, FWAH, shows a distribution that indicates much larger particles than the other hydrates and that is virtually identical to its

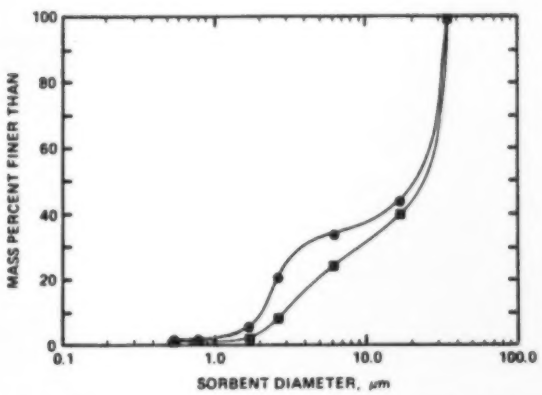

Fig. 5. Sorbent size distribution of LAH as-received (●) and size classified to < 2.0 μm (■).

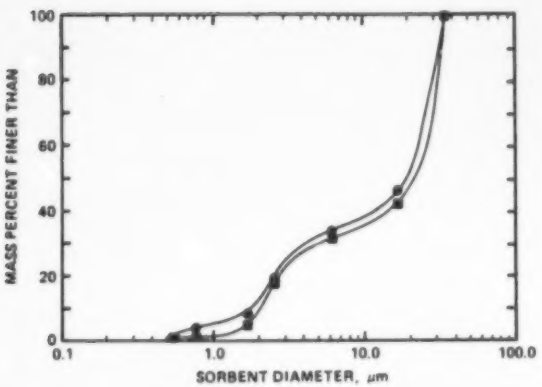

Fig. 6. Sorbent size distribution of LAH at a reactor temperature of 25 °C (●) and 1100 °C (■).

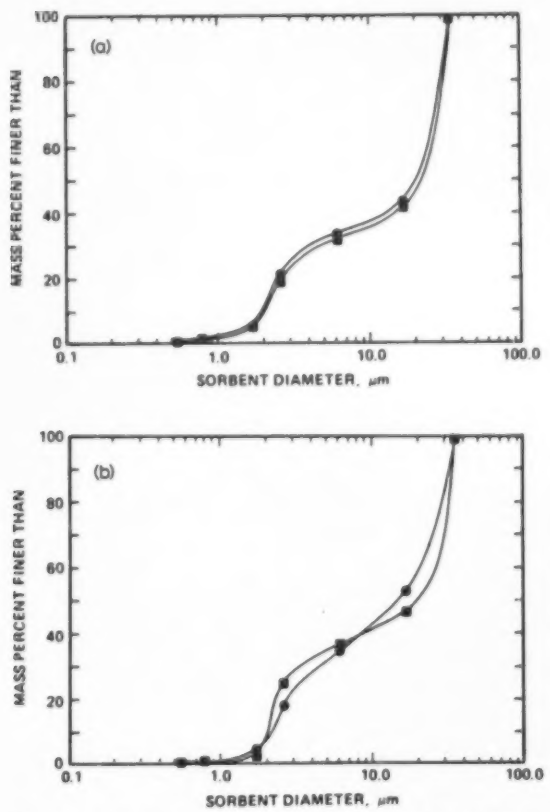

Fig. 7. (a) Sorbent size distribution of LAH with (●) and without (■) SO_2 exposure. (b) Sorbent size distribution of LPH with (●) and without (■) SO_2 exposure.

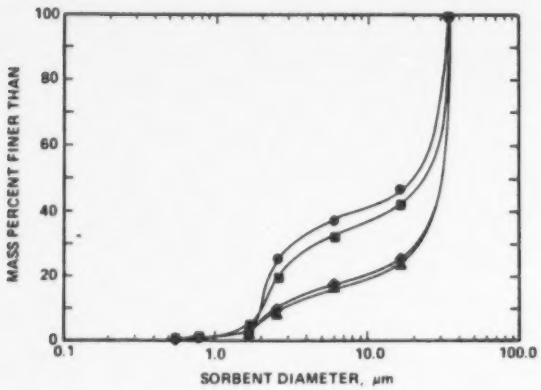

Fig. 8. Sorbent size distribution of (●) LPH, (■) LAH, (▲) FWAH, and (◆) FWL at 25°C without SO_2 exposure.

parent carbonate stone. Both of these results are surprising in that (1) hydration by slaking is known to produce finer materials than commercial methods of hydrate production, and (2) the hydration process acts to break apart the parent material, resulting in a much smaller product [5]. However, the tendency for very fine materials to agglomerate more than larger sorbents was also noted with the pre-sized hydroxide (Fig. 5). Tests with FWL did not show significant agglomeration. Aerodynamically fractionated size cuts of FWL to < 3 μm and 3–6 μm diameter were tested for subsequent sorbent size as fed onto the impactor. Little agglomeration was noted; although, the smaller particles (< 3 μm) show a greater propensity to reagglomerate than the larger ones.

Reaction testing

The conversion, X, to $CaSO_4$ by sorbent diameter for all four sorbents is shown in Fig. 9. Conversion increases with decreasing diameter for all of the sorbents. The most reactive sorbent is the slaked Fredonia, followed by both LAH and LPH, and then FWL. Previous data [4] for presized $CaCO_3$ are also plotted for purposes of comparison. Conversion changes in a linear manner with the logarithm of the sorbent diameter. This conversion increases in inverse proportion to the nth power of the sorbent diameter, d, according to:

$$\frac{X_i}{X_j} = \left(\frac{d_i}{d_j}\right)^{-n} \tag{1}$$

The values of n and the factor by which sorbent size must decrease for conversion to double, $f2$, are included in Table 2 for each of the four sorbents along with related data [4] for two calcium carbonates.

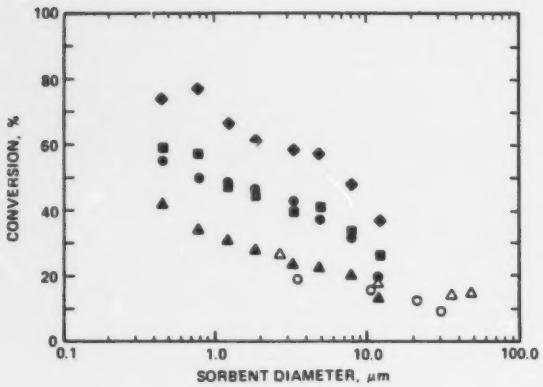

Fig. 9. Effect of sorbent diameter upon the reactivity (1100 °C, 1.5 s, 3000 ppm SO_2) of (◆) FWAH, (●) LPH, (■) LAH, and (▲) FWL compared to the results of Ishihara et al. [4] for Hikawa limestone (△) and Mie limestone (○).

The sorbent size distributions were matched up with the conversion by size to determine a composite conversion, X_c. These composite conversions were compared with values determined by collecting the whole sample, X_w, in a vial and analyzing it. Although the order of sorbent conversion is consistent (Table 3), the X_c value for FWAH is high relative to X_w, while for FWL, X_c is low relative to X_w. The hydrates' X_c values may be higher than X_w because the sorbent capture cyclone loses over 10 wt.-% of the collected sample as fines. Since these small diameter fines have high reactivities, the X_w values will be lowered. Of course, this explanation works against the discrepancy with FWL. It is likely that reactivity with increasing sorbent size becomes asymptotic. In that case, the X_c value may have been underestimated due to the large amount (ca. 75%) of sorbent mass in a range beyond the mass median diameter of the largest tray (12.1 μm) analyzed for conversion. In either case, the combined precision limits of the particle size and conversion data, plus the unexpectedly large percentage of sorbent with

TABLE 2

Values of n and $f2$ for test and comparison sorbents

Sorbent	n	$f2$
LAH	0.24	18.0
LPH	0.30	10.1
FWAH	0.22	23.4
FWL	0.32	8.7
Ref. 4	0.28	11.9

TABLE 3

Comparison of composite, X_c, and whole, X_w, conversions

Sorbent	X_c (%)	X_w (%)
LAH	26.1	23.1
LPH	24.8	21.4
FWAH	36.4	26.8
FWL	10.2	20.1

diameters in excess of the impactors' tray range, lend considerable uncertainty to the method of X_c calculation.

DISCUSSION

Fig. 3 shows that methods of testing for particle size such as gravity sedimentation do not appear to provide an accurate determination of in situ hydroxide size. This is because $Ca(OH)_2$ is extremely prone to agglomerate, regardless of temperature (Fig. 6), presence of SO_2 (Fig. 7a and b), or pre-sizing (Fig. 5).

The tendency to agglomerate extends to all three calcium hydroxide sorbents tested. Results from scanning electron microscopic (SEM) observation, gravity sedimentation, and mercury porosimetry indicate that the hydroxide agglomerates are primarily composed of constituent particles of nominal diameter less than 2 μm. Fig. 8, however, clearly indicates that the in situ sorbent size has a mass median diameter much greater than that of the constituent particles. Agglomeration is expected to occur due to interparticle attractive forces such as electrostatic charges.

The importance of in situ sorbent size upon reactivity is evident in Fig. 9. The smaller the sorbent (or agglomerate) size, the greater the extent of reaction. Agglomerates react as larger particles, decreasing reactivity. It is likely that this effect is due to the longer path length for SO_2 diffusion into larger agglomerates. The intra-agglomerate SO_2 concentration, and therefore the reactant concentration at the surface of the constituent particles, will be lower than that in the bulk fluid, making the observed, or global rate, decline. As agglomerate size decreases, the effective length of the diffusion path will shorten, decreasing intraparticle pore diffusion resistances and allowing the surface SO_2 concentration to approach that of the bulk gas. This will lead to higher reaction rates and is reflected by the higher conversion levels of the smaller agglomerates.

While FWL did not show evidence of significant agglomeration, its reactivity reflected that of the hydroxides, increasing with smaller sorbent size. This is likely due to the greater surface area to volume ratio as particle

TABLE 4

Sorbent physical characteristics and whole-sample conversion levels

Sorbent name	Conversion X_w (%)	Raw surface area (m^2/g)	Sphere diameter (μm)
FWAH	26.8	45.6	0.06
LAH	23.1	15.5	0.17
LPH	21.4	8.0	0.33
FWL	20.1	1.8	1.2

size decreases, allowing greater accessibility of SO_2 to the CaO. In larger particles, formation of a surface product layer then seals off internal surface area to SO_2, slowing the reaction.

Direct comparison of the $Ca(OH)_2$ and $CaCO_3$ results would ignore the differences inherent in their thermal decomposition (e.g., rate, temperature dependency, products) to the active form, CaO. The surface activity of CaO varies considerably, dependent upon the sorbent type, temperature and time of decomposition [8]. The water vapor evolved during decomposition of $Ca(OH)_2$ has been found to enhance the rate of crystal growth, resulting in surface area decreases of the product, CaO [9]. Similarly, the carbon dioxide evolved during calcination can affect the decomposition of proximal particles —the rate of calcination is proportional to the difference between the equilibrium partial pressure of CO_2 and the concentration of CO_2 at the sorbent surface [10]. Thus, the decomposition products may have differing deleterious effects upon the surface activity and intra-agglomerate SO_2 concentration, although the latter is not likely to extend to the SO_2 concentration at the bulk particle surface, since the sulfur to calcium ratio is about 9 : 1.

The in situ sorbent size is not the sole determinant of calcium-based sorbent reactivity. Fig. 9 clearly shows that similarly sized sorbents react to different conversion levels. For a given agglomerate size, as the size of the constituent particle decreases, the surface area to volume ratio will increase. This would delay the formation of a reaction-limiting surface product layer. When the sorbent agglomerate is idealized as being composed of uniform spheres, a theoretical sphere diameter can be determined from the specific surface area of the raw sorbent. These values can be seen in Table 4. Indeed, the order of sorbent reactivity is inversely related to the size of the agglomerates' constituent particles.

CONCLUSIONS

Testing on a limited number of calcium-based sorbents has shown that $Ca(OH)_2$ is prone to agglomerate to a much greater extent than $CaCO_3$.

These agglomerates appear to remain relatively stable in a high-temperature, reactive environment. The size of these agglomerates is significant in that the reactivity of all sorbents tested was inversely proportional to the in situ sorbent diameter over the test range of 0.44 to 12.1 μm. As sorbent size decreases, the resistance to intra-agglomerate diffusion of SO_2 declines, allowing the reaction rate to approach the intrinsic rate. Sorbents react to different conversion levels at the same sorbent diameter, indicating that the agglomerate size alone does not determine reactivity. Sorbents with the same agglomerate diameter become more reactive as the size of the constituent particles decreases with a concurrent increase in the surface area to volume ratio.

ACKNOWLEDGEMENTS

This work was supported by the U.S. Environmental Protection Agency's Air and Energy Engineering Research Laboratory. The authors are grateful to Richard Cunningham, Kevin Bruce, and Laura Beach of Acurex Corporation for their major supportive efforts.

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